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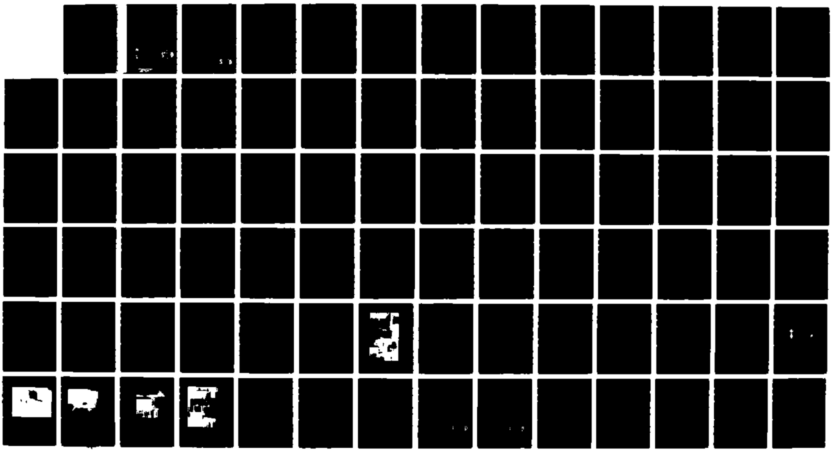

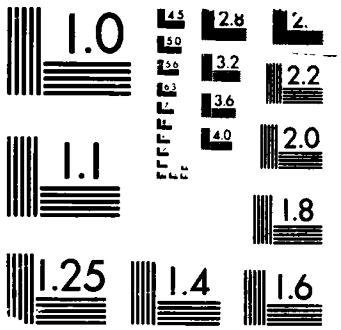

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DRES PHYSICAL CHEMISTRY LABORATORY PROCEDURES MANUAL

VOLUME II

RHEOMETRICS FLUIDS RHEOMETER

by

M.D. Gauthier-Mayer

PCN 251SC-11-A

July 1987

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ABSTRACT

The Rheometrics Fluids Rheometer (RFR) is a sophisticated, automated device for measuring the rheological properties of low to medium viscosity fluids. This publication is a detailed users manual for the RFR. It contains information on the instrumentation, daily operations and special procedures for the RFR. It should be used in conjunction with the Operations Manual for the RFR supplied by the manufacturer, Rheometrics, Inc.

ACKNOWLEDGEMENTS

The author would like to thank Dr. S.J. Armour for many helpful discussions, Mr. J. Ogston for drawing the figures and Mr. G. Poirier for testing the instructions. The help given by the technical staff of Rheometrics, Inc., who explained and detailed the various procedures was greatly appreciated.

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INTRODUCTION

The Rheometrics Fluids Rheometer (RFR) is an automated device for measuring the rheological properties of low to medium viscosity fluids (0.01 to 100 poise). It is a sophisticated, delicate instrument, and must be used with care. When Defence Research Establishment Suffield (DRES) purchased an RFR in 1980, there was no manual supplied with it. Consequently, a set of detailed notes on the use and care of the RFR was complied at DRES, through experience and discussions with the manufacturer (Rheometrics Inc.) and other users. This users manual is the end product of those notes. Rheometrics did supply an Operations Manual for the RFR (1) in 1983. The two manuals should be used in conjunction with each other.

The RFR is manufactured by Rheometrics, Inc. of Piscataway, New Jersey. The instrument can measure viscosity (η) and first normal stress difference (N_1) in steady shear rate mode and dynamic viscosity (η') and storage modulus (G') in dynamic mode. The steady shear rate range is from 0.01 to 10,000 sec^{-1} (depending on the fixture), and the dynamic range is from 0.01 to 500 rad/sec (1).

The RFR has two components - a test station and a microprocessor. The sample is measured at the test station, using one of three geometries - cone/plate, parallel plate or couette. For all three geometries, the lower fixture is a cup, with a flat bottom plate, which holds the sample. This sample cup is attached to a motor underneath it, which rotates the sample in one direction (clockwise or counter-clockwise) for steady shear, or sinusoidally for dynamic shear. The upper fixture, either a cone, plate or bob, is lowered into the sample. The torque and normal forces generated during an experiment are measured by two linear variable difference transducers (LVDT) located

above the upper fixture (100 g·cm transducer for torque and 100 g for normal force). The sample temperature is controlled by a circulating bath which surrounds the sample cup.

The RFR microprocessor is used to control the experimental parameters and to analyze and output the raw data from the test station. The sample temperature, the geometry, the type of experiment, the initial rate, the rate increments and the final rate are input through the microprocessor. The raw data (voltage from the LVDT's) is analyzed by the microprocessor and outputed on a Texas Instrument Silent 700 terminal. The output format is optional. Usually torque, normal force (N), η, N_1, and shear rate ($\dot{\gamma}$), are printed. The data can also be plotted on a built-in Hewlett-Packard X-Y recorder.

The RFR is a sophisticated, automatic rheometer. It is capable of detailed rheological testing. It is also expensive, with a price tag of ~ $200,000 (CDN). If problems occur with the microprocessor, usually a Rheometrics service person is required to diagnose and repair the instrument. This is very costly, due to the distance between DRES and New Jersey. The RFR is a delicate instrument and must be handled with care. This manual provides detailed instructions on its day to day operation.

INSTRUMENTATION

The RFR (Model 7800) consists of two main units - the test station and the microprocessor. However, there are many other pieces of equipment used with the RFR in the course of the experiment. All equipment used will be discussed in this section.

The RFR setup at DRES is shown in Figure 1. Included in the photo are the RFR test station, the RFR microprocessor, the Texas Instrument Silent 700 data terminal, a Hewlett-Packard 7132A strip chart recorder, a Micro-g vibration isolation table (under the RFR Test Station) and a Nicolet Model 206 digital oscilloscope. Not shown in the photo are the Tecam C-400 circulator, Topaz Line 2 power conditioner and a Fluke 8024A multimeter.

RFR Test Station

The RFR test station is shown schematically in Figure 2. The various parts are discussed in detail in the RFR Operations Manual (1). The test station should be placed so that it is level, and isolated from vibrations (see the vibration isolation table description below). It should also be away from drafts or severe changes in temperature.

There is a requirement for clean, dry air (or N_2) for the transducer (#2-16) and the motor (#2-15). The air attachment is at the left side of test station (#2-19). This is also where the air pulley (used in the torque calibration) is located.

The sample is loaded into the sample cup (#2-8). A suitable circulating fluid is put into the environmental chamber (#2-7) for temperature control. The connections to the circulator are shown at #2-9. An iron/constantan thermocouple, plugged into the test station at #2-5, is used to monitor the temperature of fluid in the environmental chamber. The test fixture is attached to the shaft of the transducer (#2-20). The transducer is locked in position by the spindle lock (#2-17). To move the transducer, the spindle lock is loosened and the housing is moved with the coarse spindle position (#2-3) or the fine spindle position (#2-4). For a cone and plate

experiment, the transducer and fixture are lowered into the sample to a predetermined height or gap. The gap is set, before the sample is loaded, by lowering the transducer until the gap indicator gauge (#2-1) hits the setting pen. At this point the fixture is about 2 cm from the bottom of the sample container. The transducer is lowered further until the fixture just contacts the sampler container. The coarse indicator adjust (#2-2) by the setting pen is loosened and the gap indicator is set to zero using the fine indicator adjust (#2-6). The position stop (#2-18) is used to set the gap to the desired height.

RFR Microprocessor

There are two parts to the microprocessor - the signal conditioner (Figure 3) and the computer (Figure 4). Together they allow input of the experimental details, oversee the experiment and output the data during the experiment.

The signal conditioner is the top drawer of the microprocessor. On the front panel, there are two meters (#3-5) which measure the torque and normal force output from the tranducer, from 0-100%. This is a somewhat coarse method of measuring the transducer outputs. Under the meters are the coarse and fine offset adjusts (#3-1, 3-2) for the meters, a switch to indicate which transducer is installed (#3-4) and the torque and normal force calibration screws (#3-3). The only other control that is regularly used is the dynamic/steady selector (#3-14).

The rear of the signal conditioner drawer has various connections, to the test station and the computer. Of particular note are the strain, T and Z BNC connectors. These are used to monitor the strain, torque and normal force outputs, respectively.

The computer is the bottom drawer of the microprocessor unit. The data select switches (#4-1) allow various parameters to be displayed on the digital displays (#4-2). The Bath HIGH/LOW light (#4-3) indicates whether the environmental chamber is above or below the selected temperature (the temperature can be displayed on the digital readout #4-1). The desired temperature is dialed into the TEMP thumbwheel (#4-6), in °C. The MODE switch (#4-4) is used to select the type of test desired (single, rate sweep, temperature sweep, etc.). The TEST switch (#4-5) corresponds to the type of fixture used (cone/plate, parallel plate, etc.). The RATE thumbwheel (#4-7) is for the initial rate (sec^{-1} for steady shear or rad/sec for dynamic sweeps). The STRAIN % thumbwheel (#4-8) is used to input the % strain used for a dynamic measurement. The DATA SELECT switches (#4-9, 4-10, 4-11), PLOTTER PRINTER, TEST GEOMETRY and SWEEP PARAMETERS allow the user to imput the various experiment parameters via the data terminal. The ENABLE PRINT and ENABLE PLOT switches (#4-12) allow the computer to send the calculated data to the appropriate device. The SYSTEM CONTROL switches (#4-13, 4-14) are used to control the experiment. START places the sample under the control of the computer. RESET terminates the test. HOLD interrupts the test in progress, and maintains the current rate. The READ FOR AVERAGE button is used in the steady mode, to take a measurement. The STATUS indicators (#4-15) are monitored to determine how the experiment is proceeding. The INPUT OVERLOAD indicates the transducer range or % strain has been exceeded. The MEASURE IN PROGRESS indicates when the computer is collecting data.

The rear of the computer drawer has several connections between the test station, the signal conditioner, the plotter and the data terminal. Of particular note is the S1 switch. This is the computer reset and is used when there has been a power flux or the computer

hangs up. The FUNCTION SELECT switch should also be noted. It has four positions: sine wave, ramp input, square wave and TEST. During normal system operation, the sine wave is selected, which provides a sinusoidal strain command to the strain servo. The TEST position is used during maintenance of the RFR.

Data Terminal

The data terminal is a Texas Instrument Silent 700 Model 743 KSR (2). It is used to input information to the microprocessor, and output data.

The data terminal is left on line at all times. It is plugged into the power strip at the back of the microprocessor unit, and is turned on when the main power to the RFR is turned on. The carrier detect indicator should be lit when the terminal is on. Set the speed switch to HIGH (30 characters/second), duplex to FULL (transmit and receive) and ONLINE (under computer control). When the main power to the RFR is turned on, the computer initializes and prints at the data terminal "RFR SOFTWARE VERSION 2.7".

The data terminal uses thermal paper. The paper will gradually darken when exposed to light. Any outputs should be stored in a binder for further reference. The printhead should be cleaned with alcohol, as described in reference 2, whenever the paper is changed.

Data is input to the microprocessor via the data terminal. As well, the terminal can be used as a typewriter to annotate each fluid tested. It is a good idea to type the fluid identification and experimental parameters before each run. The Silent 700 uses ASCII code. The following keystrokes will be useful when typing at the data

terminal:

a. CTRL C - if a correction is needed prior to pressing RETURN this will eliminate the entire line.

b. CTRL H - backspaces the printhead so the entry may be retyped. This is also used before the RETURN key is pressed.

c. ESC - if the RETURN key has been pressed, and an entry to a data select mode is incorrect, the ESC key will stop the inquires. The previous selections will remain in the computer memory. Restart the selections by pressing the appropriate data select key.

d. CNTL P - this initiates a new page, with heading. Otherwise a new page will automatically appear after 50 lines of print.

Plotter

The plotter, built into the RFR microprocessor unit, is a Hewlett-Packard 7010B X-Y recorder (3). It is used to plot the data during an experiment. Time, Temperature and Strain sweeps require semilog paper, while Rate sweeps require log-log paper.

Usually the plotter is used under computer control, with the NORM/AUX switch in the NORM position. The AUX position allows the Y input to be torque, normal, strain or any external input, and the X input is time.

During an experiment, each measurement is plotted as a discrete point on the graph. Several runs may be plotted on one graph paper, by using different colored pens. It is necessary to set the border limits each time the paper or pen is changed.

Strip Chart Recorder

The recorder used at DRES is a Hewlett-Packard 7132A model (4). It is very important to monitor the torque and normal force during a steady shear measurement, and this is very easily done using a strip chart recorder. As well, it provides a permanent record of the torque and normal outputs.

The recorder should be plugged into the outlet strip at the back of the microprocessor unit. The torque and normal outputs are obtained from BNC connectors at the back of the signal conditioner cabinet. The normal output will always be in the same direction, while the torque output depends on the direction of rotation of the sample cup. To compensate for the change of direction, the torque baseline should be changed, rather than switching the input leads. This makes directional changes easier to identify on the recordings.

Oscilloscope

A Nicolet Model 206 digital recording oscilloscope (5) is used with the RFR to zero the gap. The manufacturer's recommended method of setting the gap uses the normal meter on the front of the signal conditioner cabinet to determine when the fixture is touching the sample cup. However, this is a very insensitive method. The oscilloscope offers a greatly expanded scale, for more sensitive measurements.

The scope is plugged into the outlet strip at the back of the microprocessor cabinet. The normal output is taken from the BNC connector at the back of the signal conditioner cabinet. The Nicolet oscilloscope can also be used to record the normal and torque outputs during a run, via the magnetic disk drive. Operational details are contained in the Nicolet User's Manual (5).

Circulating Bath

The circulating bath supplied with the RFR is a Tecam C-400 circulator (6). It can be used from -20 to 80°C, depending on the circulating fluid used. For the normal temperature range of 20-45°C, distilled water is used.

Initially the circulator was located in the rear of the test station casting. This led to vibrational problems with the transducers. Consequently the circulator was moved away from the casting. The circulator requires a connection to cooling water for operation near room temperature. This is not needed for higher temperatures. The circulator is connected to the environmental chamber via quick connectors. These are very awkwardly placed, making the connection difficult.

When the circulator is started, there should be approximately one inch of fluid in the environmental chamber. This is enough to cover the connectors and prevent air from entering the tubing. However, the fluid volume in the chamber increases once the circulator is on and any more than an inch of fluid would cause flooding of the sample cup.

Vibration Isolation Table

The RFR test station must be isolated from vibrations which may interfere with the transducer measurements. To this end, the test station is situated on a Micro-g vibration isolator air table (7). The air table has pressurized pneumatic air springs and requires a source of clean, dry air or N_2 gas. The air table can be leveled by means of built-in leveling adjustment screws. This must be done when the air to the table is on. The table remains level through adjustable screw valves. When weight is added or removed, or the table top is bumped, the valves adjust the pressure and restore level within a few seconds.

Power Conditioner

The RFR microprocessor is very sensitive to changes in supplied power. To alleviate this, a Topaz Line 2 power conditioner (8) is used to provide clean, steady AC power. It protects against noise disturbances and voltage variations. In addition to the RFR, all other equipment attached to the RFR using electrical power must be hooked up through the power conditioner. Otherwise some line noise may be transmitted through the ground connection. This means that the oscilloscope and the strip chart recorder must be plugged into the power bar in the back of the microprocessor unit.

Multimeter

A multimeter is very useful for calibrating the torque and normal forces. If a multimeter is not available, either the oscilloscope or the strip chart recorder can be used. At DRES, a Fluke 8024A multimeter is used. The full scale deflection for the torque or normal force is 10 V DC.

RFR Accessories

There are several accessories for the RFR. These are described below.

a. Cones

The cones shown in Figure 5 have very shallow angles, 0.01 to 0.04 radians (or 0.3 to 1.1°). The tips have been truncated. When the cone is in the sample, there is a gap between the bottom of the sample cup and the cone. The gap is exactly the length of the tip that was truncated, and is usally around 50 microns. Several different cones have been supplied by the manufacturer. The early ones were made from thin aluminum, coated green, and were easily distorted. They have been replaced by ones made from titanium. Each of the titanium cones has a serial number and a calibration sheet, certifying the cone angle and the gap setting. Copies of the certification sheets for the three DRES cones (all 25 mm Radius; 0.01, 0.02, 0.04 radians) are in Appendix I. Each of the cones came in a padded wooden box. They should be stored in the boxes to prevent damage.

b. Parallel Plate

The parallel plate fixture is simply a flat plate, that is parallel to the bottom of the sample cup. As with the cones, the early parallel plates were made from aluminum, coated green. These have been replaced with a more substantial titanium parallel plate. The plate has a radius of 25 mm.

c. Couette Assembly

The couette assembly consists of a cup and a bob. These are the original fixtures supplied by the manufacturer. To date they have not been tested.

d. Calibration Fixture

The calibration fixture is a parallel plate made from aluminum, with an arm extending from the shaft, as shown in Figure 7. The arm has a notch in it exactly two centimeters from the center axis of the shaft of the fixture. Weights are hung by thread from the notch in the arm, over the air pulley. The resulting torque is equal to the mass x 2 cm.

e. Sample Cup

Two different sample cups have been supplied by the manufacturer. The first has a glass bottom and sides (see Figure 6). The second container has a polished titanium bottom and glass sides.

f. Environmental Chamber

The environmental chamber is made from plexiglass. It has three legs which mount onto the stage above the motor. It has two quick connectors which couple to the circulating bath tubing. The sample cup fits snugly in the center of the chamber. A donut-shaped rubber gasket provides a seal around the sample cup shaft and prevents leakage. This gasket must be

replaced periodically. The chamber should be cleaned with a mild soap solution only - solvents will soften or craze the plexiglass.

g. Transducers

DRES has two transducers for the RFR. One has a 100 g·cm torque range and a 100 g normal force range. The other transducer (modified from a 10g·cm torque range and a 100 g normal force range) has a 1000 g·cm torque range and a 1000 g normal force range. Each transducer has its own torque and normal circuit boards. The transducers are not readily interchanged. The process of installation, alignment, electrical and weight calibration is tedious and can take up to a week to complete.

h. Extender Boards

There are two extender boards for the RFR, one for the small circuit boards in the signal conditioner drawer, and one for the larger circuit boards in the computer drawer. These are necessary for some of the electrical calibrations.

i. Spare Boards

DRES has purchased several spare circiut boards, to be used if the working ones fail. The ones on hand are:

- A/D Converter Board
- Frequency Synthesizer
- Computer Board
- Temperature board
- Signal Conditioner board

j. Dial Indicator Gauge

The dial indicator gauge is used to determine whether the transducer shaft is aligned with the motor shaft with respect to concentricity and flatness. The gauge is mounted in the motor shaft (after removing the environmental chamber and sample cup); and the dial rests on the edge of a parallel plate fixture (or the calibration fixture) (See Figure 9). The gauge is turned the full circle of the fixture and the change in the dial reading is recorded. The mirror mounted behind the motor shaft is very useful in reading the gauge when it is facing the rear.

k. Alignment Fixture (made at DRES)

The alignment fixture is a hollow tube which fits into the motor shaft. The shaft of the transducer just fits into the opening of the tube. It is used to roughly align the transducer to the motor shaft, when the transducer is first installed.

DATA REDUCTION

The raw data is automatically reduced by the microprocessor. The equations used by the RFR are given in Reference 1.

DAILY OPERATIONS

Note: The instrument must be on for a minimum of 30 minutes before any operations are performed.

1. Air Table

   Ensure the air table has a source of clean, dry air or N_2 gas.

For the N_2 cylinder, set the regulator to 50 PSI. This gives a reading of 25 on the gauge under the air table.

2. Transducer

Ensure the airbearing in the transducer has a supply of clean, dry air or N_2 gas. For the N_2 cylinder, set the regulator to 45 PSI. Any moisture or dust in the air supply will damage the transducer.

3. Calibrations

Both the normal and torque outputs must be calibrated daily. The readings should be recorded, along with the associated voltages, on a form such as that shown in Appendix II. Normally, the torque is calibrated first, then the normal force.

a. Torque (See Figure 7)

1) Place the calibration plate on the transducer shaft, with the calibration arm facing forward.

2) Install the air pulley at the left side of the transducer shaft as in Figure 7. Turn on the air to the air pulley, until the arms of the pulley rotate very slowly (only a slight air pressure is needed).

3) Set the instrument according to the settings listed in Table I until the "Initiate" setting. Be sure the environmental chamber is off, as the temperature is set to 700°. Monitor the torque output from the T BNC at the back of the signal conditioner drawer, with the voltmeter.

Note: 100 g·cm = 10 V (maximum output)

4) Zero the torque meter on the front of the microprocessor (#3-5) using the fine offset adjust (#3-2), so the needle is at zero percent. This is easily done using the voltmeter. The offset adjusts for the meter are not used once a calibration begins. However, the fine offset may be used in between two calibrations.

5) Depress the START, HOLD switches. The motor will turn the sample cup at the rate input. The readings are done automatically and the values appear on the TORQ display.

6) Start at 0 g with no weights on the arm. The value torque should be less than 0.2% of full scale (i.e., ≤ 0.2 g·cm for a 100 g·cm transducer). Record the TORQ result and the voltage. The zero reading is very important. The computer uses the voltage measurement at zero grams to calculate an electronic offset that is applied to all other measurements. If a low zero is not obtained, press RESET and start again.

Note: Wait until two or three measurements have been taken by the RFR (as indicated by the MEASUREMENT IN PROGRESS indicator) before recording the torque value. The initial reading may not be representative.

7) When the MEASURE IN PROGRESS light is off, carefully hang a 20 g weight from the calibration arm of the calibration plate, over the front arm of the air pulley. Be sure the weight does not oscillate, or touch the air pulley or the water in the environmental chamber. Record the reading and the voltage (should be ∿ 40 g·cm and 4V).

8) Repeat step 7, using 40 g (80 g·cm). The actual calibration should be done at this weight, if the voltages are

not as expected. Adjust the fine calibration screw (#3-3) under the torque meter, until the TORQ display reads 80.0 g·cm.

9) Press RESET, wait 10 seconds, then repeat the calibration procedure from step 4.

Note: The transducer should give linear, repeatable results.

10) If the readings are consistent, then the torque output is calibrated. Turn off the air pulley and remove it.

b. Normal (See Figure 8)

1) Set the instrument according to Table I until the "Initiate" setting. The calibration plate used for the torque calibration is also used for the normal calibration.

Note: Be sure to change the temperature thumbwheel to 25°C before turning on the environmental chamber.

2) Ensure the NORMAL meter on the front of the microprocessor (#3-5) is zeroed. Use the voltmeter and the fine offset adjust (#3-2) under the meter. This offset adjust is not used once a calibration has started, but may be adjusted after the RESET has been pressed, before the next calibration. The normal signal is monitored from the Z BNC at the back of the signal conditioner drawer.

3) Start the calibration without any weights. Press START and HOLD. In steady mode, the readings must be taken manually, by pressing the READ FOR AVERAGE switch. Wait until the normal force is constant before starting the reading. Take the reading for ∿ 10 sec., then press the READ FOR AVERAGE.

switch again. The normal force will appear on the NORM display. Record the value and the voltage.

4) Place the weights for the next reading directly on the top of the calibration plate. The weights should be placed symmetrically around the shaft, as close to the shaft as possible.

5) Readings should be done at 40 g (use 2 x 20 g), 60 g (use 3 x 20 g) and 80 g (use 4 x 20 g). Calibrate at 80 g, if any adjustments are necessary. Adjust the fine calibration screw (#3-3) under the NORMAL meter, until the NORMAL display reads 80.00 g.

6) Press RESET, wait 10 seconds, then repeat the calibration procedure, from step 2.

Note: Usually the normal is much harder to calibrate than the torque. If the normal appears calibrated on the first calibration sequence, then often it is not correct on the second calibration sequence, after the instrument has been reset. The sequence may have to be repeated 3-4 times, before consistent values are obtained.

The instrument is now calibrated and is ready for a sample run. Remove the calibration fixture.

4. Zeroing the Gap (for cone/plate or parallel plate fixtures)

Install the fixture required for the experiment. Everytime a fixture (a cone or the parallel plate) is installed, the gap zero must be checked. The gap setting is very important in obtaining accurate results. The gap zero is set using the Nicolet digital oscilloscope and the normal output (from the Z BNC, at the back of the signal

conditioner drawer). This is a delicate procedure and should be done with care. Otherwise, damage will result to the transducer.

a. Oscilloscope Settings
   ON
   LIVE input
   10 μ sec/point
   auto mode
   1 volt x 1 scale
   auto center off
   Adjust the signal on the scope, so that it is sensitive and is centered on the horizontal cross hair.

b. Unlock the transducer, using the spindle lock (#2-17) at the left front of the test station.

c. Use the coarse spindle position (#2-3) on the right hand side of the casting, to lower the transducer housing (the gear lock by this knob should be in the bottom position). Stop the motion when the gap indicator gauge (#2-1) makes contact with the setting pin.

Note: Be sure to watch the fixture as it is being lowered. At this point, it should be ∿ 2 cm above the bottom of the sample cup.

d. Lock the transducer in place. Shift the gear lock (by the coarse spindle adjust) to the top position. This engages the fine spindle adjust (#2-4). Unlock the transducer.

e. Slowly lower the transducer using the fine spindle adjust, until the fixture just touches the sample container, as

indicated by a large change in the normal force output on the scope.

f. Back off slightly. Again, slowly lower the plate, until the normal signal just barely moves. This is the zero position.

g. Now set the gap indicator gauge to zero, using the fine indicator adjust (#2-6).

h. Repeat f and g until the gap is correctly set to zero. At this point, the fine indicator adjust should be tight.

5. Setting the Gap

For cone/plate measurements, the gap must be set to a specific value, according to the calibration sheet accompanying each cone. This is not necessary for parallel plate measurements, since the gap setting is variable in this mode. The gap is set before the sample is loaded into the sample container.

a. Raise the transducer until the gap indicator gauge reads the necessary gap measurement plus 4 microns. That is, for a 50 μ gap, set the instrument to 54 μ. (The 4 microns is the slack in the movement.)

b. Insert the feeler gauge strip at the position stop screw (#2-18). Mark this spot on the feeler gauge strip using a felt pen.

Note: The feeler gauge strip is 2/1000 in., but any feeler gauge may be used.

c. Gently lower the position stop screw onto the feeler gauge. Then lock the screw in place with the locking nut. The gap indicator gauge should not have moved.

d. Raise the transducer. Lower it down until the stop screw hits the feeler gauge. The gap indicator gauge should read the desired value (i.e., 50 μ). Otherwise, repeat steps a-c.

e. Repeat step d several times, until the gap indicator gauge consistently reads the correct value.

f. Raise the transducer, and remove the piece of feeler gauge.

The instrument is ready for the sample.

6. Loading the Sample

The size of the sample will vary, depending on the type and size of fixtures used. A listing of the volumes needed for the various fixtures is in Table II. The easiest way to measure and insert a sample is with a disposable syringe. For viscous material, care must be taken to fill the syringe slowly. Otherwise bubbles will be introduced. The syringes are used once, then discarded in the chemical waste jars, to prevent any sample contamination.

a. Raise the transducer housing.

b. Slowly empty the syringe into the centre of the sample cup.

c. Allow any bubbles to rise out of the sample.

d. For low viscosity fluids ($\leq$ 15 p) it will be necessary to lower the cone/plate fixture immediately, before the sample runs to the edge of the container. For more viscous fluids (> 15 p), the sample can be left for 10-15 minutes before lowering the fixture, to ensure all bubbles are out of the sample.

e. Slowly lower the transducer assembly.

Note: 1. For cone/plate measurements the feeler gauge should be in position so that when the position stop screw hits the feeler gauge, the fixture will be at the correct gap setting.

2. For parallel plate measurements, lower the fixture until the normal force just starts to register on the normal force meter. Record the gap from the gap indicator gauge.

f. With the motor off, slowly turn the cup by hand, ensuring that the fluid completely fills the area under the fixture. There should not be any sample on top of the fixture.

g. Allow the sample to come to temperature equilibrium for 10-15 minutes before any measurements are made. For viscoelastic materials, the normal force must be monitored during this time. The material must be fully relaxed (constant normal force reading on the strip chart recorder) before testing can begin.

7. Data Collection

There are many measurement options available on the RFR. The user can choose to do a single rate or a rate sweep. A temperature sweep at one rate may be chosen or a combination frequency/temperature sweep. In the dynamic measurements a strain sweep can be chosen. There is also a cure mode, transient mode and thixotropic loop mode. In general, testing is done in steady or dynamic shear with a rate sweep. The single rate measurement is useful too, for rapid sample evaluation at the start of an experiment. The temperatures and strains are usually changed manually, rather than using the preprogrammed steps in the RFR. The different options are described in detail in Chapter

one of Reference 1. Typical steady shear and dynamic mode experiments will be described in the next two sections.

8. Steady Shear Experiment

Measurements are made in either the clockwise (CW) or counter-clockwise (CCW) direction. Usually the sample is tested over a range of shear rates in one direction, allowed to relax for several minutes then measured at the same rates in the opposite direction. The following selections must be determined for a steady shear measurement.

a. Direction - choose STEADY CW or STEADY CCW
Be sure to note the selection on the printer and strip chart outputs.

b. Mode - Select RATE SWEEP (or SINGLE)

c. Dial in the initial rate (in sec^{-1}) on the thumbwheel. The initial rate should be large enough to give an initial torque value of one percent of full scale (i.e. 1 g·cm for a 100 g·cm transducer). If a rate sweep has been selected, then press SWEEP PARAMETERS and input the following data on the data terminal:

1) Last Rate - the maximum rate for the run. The final rate should be such that the torque or normal forces are still on scale.

2) Points per decade - usually there are 10 points per decade. The rates are divided up logarithmically.

3) Readings per rate There is only one reading per rate, when the measurements are in the same direction. If CW and CCW measurements were taken and averaged together at each rate, then there would be two readings per rate.

d. TEST Selection - select the fixture used (cone/plate or parallel plate). Then press TEST GEOMETRY and enter the fixture measurements via the data terminal.

1) Cone/Plate - enter radius and cone angle.

2) Parallel Plate - enter gap and radius.

3) Couette - enter cup radius, bob radius and bob length.

e. Temperature - Dial in the desired temperature (in °C) using the thumbwheel. Turn on the cooling water to the fluid circulator. Ensure there is ∿ one inch of water (or other fluid) in the bath container (just enough to cover the circulation ports). At the test station, press the ENVIRONMENTAL CHAMBER switch on. Press and hold the PUMP RESET switch until the bath fluid is visibly circulating in the bath container. Monitor the temperature using the TEMP digital display, and an independent thermometer.

f. Printer - to select the printer output format press PRINTER and enter either Y (yes) or N (no) after each entry on the data terminal. The selections for output are:

1) Eta - Y
2) N_1 - Y

3) Torque - Y

4) Normal - Y

5) Temperature - Y

6) Time - N

7) Rate - Y

8) Page Title - input the current date, usually, for the page heading

Note: Only six columns are allowed. If more than six are selected, only the first six are printed out.

Next, press ENABLE PRINT, for the microprocessor to pass information to the data terminal.

g. Plots - Plots can be output during a run. It is a good idea to wait until a preliminary run has been done, so that the values of η, N_1 and rate are known before doing a plot. The output is either a semi-log or log-log plot, depending on the selections made. To select the plotter options, press PLOTTER and input the correct answers to the following questions on the data terminal.

Note: The number of cycles input for a log axis must correspond to the number of cycles on the graph paper.

1) X-axis = Temp N
   = Rate Y - usually the rate is selected for the X-axis

2) X-axis zero

3) X-axis max - select 0.1, 1, 10 etc.

4) Y-axis zero

5) Y-axis max

6) Plot Eta Y
7) Plot N_1 Y

Next, press ENABLE PLOT so that the data will be supplied to the plotter.

h. Plotter - The plotter must be initialized before any plots can be obtained. The paper used depends on the plots required. For steady measurements, semi-log paper is required for time or temperature on the X axis, and log-log paper is used for rate on the X axis. The plotter is initialized as follows.

1) press ON LINE switch
2) press SERVO on
3) place paper on the platen
4) press CHART HOLD
5) put a pen in the holder
6) ensure the switch below the platen is in the NORM position

The axes are set and zeroed by using the knobs to the left of the platen and vernier pots and push buttons below the platen.

1) minimum X - depress and hold the X button below the platen and adjust with the X knob beside the platen
2) maximum X - adjust with the X vernier pot
3) minimum Y - depress and hold the Y button below the platen and adjust the Y knob beside the platen
4) maximum Y - adjust with the Y vernier pot

i. Strip Chart Recorder - the strip chart should be monitored

from the time the fixture is lowered onto the sample until the end of the experiment. Both torque and normal force should be output.

1) Attach the outputs from the Z BNC (normal force) and the T BNC (torque) form the back of the signal conditioner to the strip chart recorder.
2) Start the recorder at a slow rate.
3) Zero the T and Z outputs while the fluid is at rest.

j. Measurements - Type into the data terminal the name of the sample fluid, the volume used, the fixture measurements and any other relevant data. Label the strip chart recording and plot paper. Monitor the sample Z output on the strip chart. If it is constant, then a measurement can be taken.

1) Ensure the motor is turned on at the test station.
2) Press START. The motor will start at the first rate. Monitor the torque digital readout. It must be ≥ 1% of full scale for a valid measurement.
3) Monitor the torque and normal outputs on the strip chart recorder. Once they are both at a constant value, press READ FOR AVERAGE. The MEASURE IN PROGRESS light will come on. Measure the sample for a minimum of three revolutions of the sample cup. Press the READ FOR AVERAGE button again. Once the MEASURE IN PROGRESS light goes out, the rate will automatically increase for a rate sweep and the data for the first point will be printed at the data terminal. If a single point is selected then release the START button, and the values will be printed.
4) For a rate sweep, repeat step 3 until the sweep is completed.

Note: If either torque or normal force goes beyond 100% of full scale, the OVERLOAD light will come on. The experiment must be stopped at this point by depressing the RESET button.

If the rate is too fast, then some of the sample will spin out of the gap, and the data will be incorrect. Again, stop the experiment by depressing the RESET button.

5) At the end of the rate sweep, allow the torque and normal force to relax to constant values.
6) Select the opposite direction, and repeat steps 2 to 5.

Note: The torque output on the strip chart recorder is now in the opposite direction and will go off the paper unless the zero is shifted.

Once measurements in the two directions are completed, a dynamic sweep may be tried. Again, the sample should have relaxed in both torque and normal force before any measurements are attempted.

9. Dynamic Shear Experiment

In a dynamic experiment, the sample cup is oscillated at a selected frequency and amplitude. Usually the sample is tested over a range of frequencies at one amplitude (strain), then the amplitude is changed, and the same rates are repeated. The following selections must be made for a dynamic shear experiment.

a. Direction - select DYNAMIC

b. Mode - select RATE SWEEP (or SINGLE)

c. Rate - dial in the initial rate (in radians/sec) on the thumbwheel. The initial rate should be large enough to give an initial torque reading of one percent of full scale. If a rate sweep has been selected, then press SWEEP PARAMETERS and input the following data on the Data Terminal.

1) last rate - the maximum rate should not exceed 100 radians/sec.
2) points per decade - usually there are 10 points per decade. The rates are divided up logarithmically.

d. TEST Selection - this is the same as described in the Steady Shear Experiment.

e. Temperature - this is the same as described in the Steady Shear Experiment.

f. Printer - To select the printer output, format, press PRINTER and enter either Y (yes) or N (no) after each entry on the data terminal. The selections for output are:

1.	G'	Y
2.	G"	Y
3.	Eta*	Y
4.	G*	N
5.	Tan delta	N
6.	Eta'	N
7.	Eta"	N
8.	Time	N

9) Rate Y
10) Strain Y
11) Temperature Y
12) Torque Y
13) Page title - input the current date for the page heading

Note: Only six columns are allowed. However, the strain is printed at the start of the output, and is not considered a column.

Next, press ENABLE PRINT, for the microprocessor to pass information to the printer.

g. Plots - The plotting proceedure is similar to that described in the Steady Shear Experiment. The plotter options are selected by pressing PLOTTER and answering the following questions at the data terminal.

1)	X - axis =	Temp	N	
	X =	Rate	Y	
	X =	Strain	N	
2)	X - axis	zero		
3)	X - axis	max		Select 0.1, 1, 10 etc.
4)	Y - axis	zero		
5)	Y - axis	max		
6)	Plot	G'	Y	
7)	Plot	G"	Y	
8)	Plot	Eta*	Y	
9)	Plot	G*	N	
10)	Plot	1/Tan Del	N	

Note: Only three options can be plotted at one time.

Next, press ENABLE PLOT so that the data is sent to the plotter.

h. Plotter - the plotter set up is as described in the steady shear experiment.

i. Strip Chart Recorder - the strip chart is NOT used for dynamic measurements. The pen would not be able to follow the oscillatory motions.

j. Measurements - type into the data terminal the name of the sample fluid, the volume used, the fixture measurements and any other relevant information. Label the plot paper. Ensure the strip chart recorder is off. Measurements are taken as follows:

1) Ensure the motor is turned on at the test station.
2) Press START. The experiment is now under the control of the computer. The measurements are done automatically at each rate setting, and printed at the data terminal.
3) Monitor the torque that is printed after each measurement. If it is greater than 75% of full scale, then stop the experiment, as the torque at the next rate will be off scale. The experiment is stopped using the RESET button.

At the end of the dynamic sweep, the strain rate may be changed, and the experiment repeated. It is recommended that the rate sweeps at different strains be plotted on one graph paper for ease of comparison.

Note: The data for two different strains (say 80% and 90%) should be the same. Otherwise choose a smaller strain, until the data is repeatable.

10. Clean Up

At the end of an experiment the following procedures should be followed.

a. Turn off the motor

b. Raise the fixture from the sample

c. Carefully remove the fixture and clean it with an appropriate solvent. Put it away in the proper wooden box.

d. Clean the sample from the sample cup. This is normally done in situ. If the sample cup must be removed for cleaning, the circulating bath must be turned off and the enviornmental chamber drained.

e. Remove the printer output. The pages should be labeled in order, then placed in a binder. The strip chart output should be labelled with the date and sample material. It should then be folded, placed into an envelop and filed along with the printer output, and any plots.

11. Shut Down Procedures

At the end of the day the following procedures should be followed:

a. Ensure the Clean Up procedures (above) have been followed.

b. Turn off the environmental chamber.

c. Turn off the cooling water to the circulating bath.

d. Turn off the air supply to the RFR.

e. Turn off the air supply to the air table.

f. Turn off the MAIN power switch, if the RFR will be inactive for more than 24 hours.

SPECIAL PROCEDURES

1. Removing a Transducer

Note: this must be done with the air supply to the RFR on.

- remove the dust cover at the top of the tranducer shaft (6 screws)
- raise the transducer
- remove the two screws which hold the cables in place along the left hand side, at the top of the transducer
- disconnect the torque and normal electrical cables
- loosen the 4 outside screws on the black base plate of the transducer (on the bottom end)
- holding the transducer gently, remove these screws

- remove the transducer from the bottom, feeding the air hose through the shaft
- shut off the air supply
- remove the air hose from the transducer

2. Installing a Transducer

There are several steps to installing a transducer - changing the torque and normal boards, ensuring the transducer cores are centered, physically installing the transducer, zeroing the transducer, calibrating the torque and normal force and setting the notch filters.

a. Each transducer has a set of torque and normal boards. When a new transducer is installed, the proper set of boards must also be installed.
   - turn the main power off
   - change the torque and normal boards in the signal conditioner drawer
   - the torque is the second last board and the normal is the last board
   - turn the main power on

b. The transducer must be checked to ensure that the cores of the LVDTs are centered in the coil housing, before the transducer is installed in the RFR.
   - set up a ring stand at the left side of test station
   - carefully place the transducer in the ring stand
   - attach the electrical connectors from the transducer to the test station.

Note: the wires to the connectors are very fragile and easily broken.

- attach a voltmeter to the computer side of the normal connector, at the red and black wires
- the voltage should be zero, if the core is centered lengthwise in the coil housing; the normal LVDT is at the top of the transducer assembly
- if the voltage is not zero, loosen the allen screw holding the coil housing in place and slowly move it up and down until the voltage is zero, at the null position
- ensure the core is also centered crosswise in the coil housing
- tighten the screw holding the coil housing
- repeat the process for the torque LVDT, which is located at the side of the transducer assembly.

c. - zero the torque meter using the coarse and fine offset adjusts (#3-1,2); if there is not enough adjustment possible, center both the adjusts, and use pot R24 on the torque board to zero the meter
- zero the normal meter in the same manner, using pot R24 on the normal board, if necessary.

d. To install the tranducer in the test station:
- disconnect the torque and normal electrical connectors
- remove the transducer from the ring stand
- attach the air supply
- turn on the air supply
- feed the transducer into the housing from the bottom
- ensure the air hose is not pinched
- holding the bottom of the transducer gently, replace the four screws which hold the transducer base plate to the housing

- reconnect the torque and normal electrical connectors
- connect the cable holder to the left side of the transducer housing
- replace the dust cover.

e. Roughly align the transducer using the alignment tool.
   - drain and remove the environmental chamber and the sample cup
   - install the alignment tool in the motor shaft
   - loosen the four screws in the bottom of the transducer base plate, slightly
   - gently lower the transducer shaft into the alignment tool, shifting the transducer so it is symmetric in the tool
   - tighten the four screws on the transducer base
   - raise the transducer.

f. When a transducer is installed the transducer shaft must be checked for flatness and concentricity relative to the motor shaft. (see Figure 9)
   - install the aluminum calibration fixture (or other flat, rigid fixture)
   - drain and remove the environmental chamber, and the sample cup
   - install the dial indicator gauge in the motor shaft
   - to check the concentricity, position the dial gauge to the outer edge of the parallel plate, and revolve the gauge around the plate. The mirror behind the motor shaft is very useful for reading the gauge.
   - slightly loosen the four outside screws, on the bottom of the transducer base plate

- tap gently on the base plate to correct misalignments
- slowly tighten the four screws, checking the concentricity to ensure that it doesn't change.

Note: the change in concentricity around the plate should be less than 2/1000 of an inch or 0.05 mm.

- check the parallel plate for flatness, all around the bottom, as close to the edge as possible
- adjustments are made at the steel base of the cover over the motor (see Figure 2, #15). Loosen the three large screws and adjust the flatness using the 3 small screws

Note: the plate should be flat all around to with 3/10000 of an inch, or 0.0076 mm

- recheck and adjust the concentricity, then the flatness until they are both within the limits

Note: accurate geometry is essential for good measurements

Note: if there are severe difficulties with the alignment, see item 9 of this section.

g. - calibrate the torque using the torque calibration board (3rd board from the front, signal conditioner drawer)
- first, turn the torque calibration screw (#3-3) fully to one end. Then turn back five full turns to get to the middle (10 turn potentiometer)
- set the RFR up for a calibration as outlined in the <u>DAILY OPERATIONS</u> section of this manual

- ensure the torque meter is at zero when there are no weights on the calibration arm
- hang a weight on the calibration arm
- adjust the correct gain pot, according to Table III, to obtain the correct reading on the torque meter and the multimeter

h. - in a similar manner, calibrate the normal force using the normal calibration board (4th board from front, signal conditioner drawer) and the normal force calibration method outlined in the DAILY OPERATIONS section. Select the correct gain pot to adjust from Table III.

2. Cross Talk Adjustment

A 10P Newtonian fluid is used when checking the cross talk between the torque and normal transducers. This is done after the torque and normal force are calibrated.

a. Turn the RFR off. Open the signal conditioner drawer, remove the torque board (2nd last card), put it on the small extender board, and place the extender board back in the drawer

b. Turn the RFR on. Set the geometry to the correct radius and cone angle.

c. Load the sample and lower the fixture.

d. Connect the output from the normal BNC connector (at the rear of the signal conditioner drawer) to the voltmeter.

e. Zero the normal force meter on the front of the signal conditioner.

f. Set the RFR to SINGLE, STEADY CW, initial rate 200 sec^{-1}. Press START, read the normal force display and voltage in the CW direction, then the CCW direction (the torque should be ~ 79 g·cm).

g. Minimize the normal force output in both directions by adjusting R3 on the torque board.

h. When the calibration is complete, turn the RFR off, remove the extender board, reinstall the torque board and close the top drawer.

3. Temperature Calibration

a. PRT Setpoint

- this is used if the thermocouple reading is correct, but the bath temperature is not what was dialed in the thumbwheel
- use the temperature board (first card on the right side, Computer drawer)
- dial in 30° with the TEMP thumbwheel
- monitor the temperature on the digital display, after the BATH HI/LOW indicator lights are out
- if the display doesn't read 30.0°, adjust pot R25 on the temperature board until the correct BATH HI/LOW indicator light is on (turn CCW to increase the temperature reading)

- repeat until the temperature reads 30.0° with both lights out
- set the temperature to 80.0° with the thumbwheel, and monitor the temperature display and the high/low indicator lights
- adjust pot R33 if necessary
- ensure the reading at 80.0° is correct, then repeat the high and low temperature adjustments until the readings are correct at 30.0° and 80.0°

b. Thermocouple Calibration

- use ice water (or crushed ice), boiling water and an accurate thermometer to calibrate the thermocouple
- plug the thermocouple into the RFR test station and insert it into the ice water
- read the TEMP digital display and the thermometer reading

Note: the thermometer reading must be corrected for the stem area above the fluid. This is described in Reference 9.

- if the TEMP display is not the same as the corrected thermometer reading, adjust pot R14 on the temperature board (first card on the right, computer drawer)
- insert the thermocouple into the boiling water. Wait until the temperature is 93°C. Make any adjustments to pot R33 on the temperature board if the TEMP display doesn't read 93°C.
- repeat using the ice water and boiling water until the display is correct at both ends of the temperature scale

Note: when the thermocouple is disconnected from the RFR test station, the TEMP digital display shows the temperature inside the microprocessor chassis.

4. Steady Rate and Balance Calibration

This calibration is done in three steps - zeroing the rate, adjusting the symmetry between the CW and CCW directions, and calibrating the rates.

a. The electronics are set to zero first
   - turn the MAIN power off
   - place the steady board (2nd from front, signal conditioner drawer) on the small extender board
   - with a voltmeter, measure the voltage between R3 (right hand side) and ground (bottom of C3)
   - dial in 0.0 on the RATE thumbwheel
   - measure the voltage with the motor off; it should be very small (< 0.01v)
   - adjust the voltage to zero on the Analog I/O board (2nd board from right in the computer drawer) at R4
   - the zero voltage will affect the drift in the motor speed.
b. Next the electrical symmetry between the CW and CCW directions is checked.
   - dial in 9.99 sec^{-1} on the rate thumbwheel (the motor is still off)
   - set the instrument to STEADY CW
   - press START
   - the voltmeter should read $\sim$ 10 V
   - reverse the direction to STEADY CCW

- the voltage should be the same value as in the CW direction, but with opposite polarity
- adjust R3 on the Analog I/O board (second from left, computer drawer) until the voltages from the two directions are the same absolute value.

c. Once the computer has been electrically calibrated (as in a and b), the rate can be checked.

- on the steady board (2nd from front, signal conditioner drawer) connect the voltmeter across C5 (or the bottom of R3 and the left side of R18). This measures the TACH voltage from the motor
- set the RFR to cone/plate geometry, 1 radian cone, STEADY CW
- input the rate as 0.628 sec^{-1}. At 1 radian cone angle, the time for one revolution should be 10.0 seconds
- position a pointer on the motor shaft, and a marker on the base plate under the pointer
- turn the motor on
- press START
- with a stop watch, measure the time for one revolution. If the time is not exactly 10.0 seconds, then adjust R10 on the steady board
- the timing must be done precisely and accurately for a good rate calibration
- note the voltage, after the rate has been properly set; this voltage should be ∿ 0.20 volts and is used to calibrate the other rates
- press RESET
- check and adjust the voltages at the following rates:

6.28 sec^{-1}	2.0V	pot R7
62.8	20.0	pot R5
0.0628	0.020	pot R13
0.00628	0.0020	pot R15

5. Dynamic Amplitude Check

a. - remove the environmental chamber and the sample cup
- install a sheet of polar graph paper, with calibration marks at -0.5, 0 and 0.5 radians
- attach the pointer to the motor shaft.

b. - attach the voltmeter to the STRAIN BNC, at the rear of the signal conditioner drawer
- in the dynamic mode, the voltage with the pointer at the 0.5 radian mark on the left side should equal the voltage at the right 0.5 radian mark.

c. - make adjustments at R37 on the first board in the signal conditioner drawer

d. - unhook the STRAIN connection between the computer and the signal conditioner
- set the RFR to 700 on the TEMP thumbwheel, 50% strain, rate of 0.2 rad/sec
- press START; the pointer should oscillate between the two 0.5 radian marks on the polar graph paper.

e. - make any adjustments to R5 on the first board of the signal conditioner drawer.

6. Accessing the Computer Memory

a. - at the back of the microprocessor, set the mode switch to TEST
- depress the TIME and BLANK displays
- type CTRL S at the data terminal and a beep will sound (S is for start)
- type in the required computer location (all locations start with D)
- type in CTRL E (to end the location)
- the readout on the display indicates what is stored in that computer location.

b. - the phase angle locator is D4ØØØ

c. - the temperature locator is D4ØFD

d. - the strain locator is D41ØØ
- ignore the decimal place in this display

e. - the frequency locator is D4ØFA

f. - the mode locator is D4ØEF
- there is a binary readout on the display which corresponds to the following:

1 = signal
2 = rate sweep
4 = temperature sweep
8 = frequency/temperature sweep
16 = strain sweep
32 = thixotropic loop

68 = cure cycle
128 = transient

g. - the geometry locator is D40F0
- the binary readout on the display corresponds to the following:
  - 1 = cone and plate
  - 2 = parallel plate
  - 4 = torsion
  - 8 = couette
  - 16 = coni-cylindrical

h. - the transducer locator is D40F7
- the binary readouts correspond to:
  - 64 = 1 g·cm
  - 32 = 10 g·cm
  - 128 = 100 g·cm

i. - the directional locator is D40F8
- the binary code corresponds to:
  - 1 = dynamic
  - 2 = steady CW
  - 4 = steady CCW

Note: be sure to turn the switch to SINE at the back of the microprocessor unit when the test is complete.

7. Phase Angle Adjustment

The phase angle is checked using a 10P Newtonian standard fluid.

a. - set the instrument to DYNAMIC, rate sweep, 30% strain
- lower the fixture onto the sample.

b. - at the back of the microprocessor, turn to the TEST mode
- depress the TIME and BLANK displays
- access the computer memory with the code D4ØØØ.

c. - press START and monitor the BLANK display
- the display should read -90°
- if there is more than a half degree difference, adjustments are made on the torque calibration board (third card from front, signal conditioner drawer).

d. - set the instrument to 80 rad/sec, SINGLE, 100% strain
- adjust pot R15 (for the 100 g·cm transducer) or pot R7 (10 g·cm transducer) until the phase angle is 90°.

e. - recheck the phase angle with a rate sweep.

8. Spikes in the Torque or Normal Outputs

Spikes which occur in the torque or normal outputs, at regular intervals may result from carbon deposits on the motor brushes. The carbon deposits may also result in an imbalance between the CW and CCW directions.

a. - set the instrument to STEADY CW, 1.0 radian cone angle, 90 sec^{-1} rate (this is 90% of the maximum rate, with this cone angle).

b. - press START, HOLD
- leave at this rate for 20 minutes.

c. - change to steady CCW, and repeat for another 20 minutes. This should eliminate the spikes.

d. - the buildup will occur if the dynamic mode is used regularly, or if using only low speeds in the steady mode
- the proceedure may need to be done on a regular, monthly basis.

9. Flatness and Concentricity Problems

Sometimes the alignment of flatness and concentricity is very difficult. There are three possible sources of error; severe initial misalignment, worn screws on the motor base plate or failure to tighten all the screws sufficiently.

a. - if the initial alignment is very bad, there may not be enough movement in the alignment screws to correct it.
- the transducer base should readily move up and down in the transucer housing. If it doesn't, loosen the four screws in the baseplate and reposition the transducer.
- use the alignment tool to get a good initial alignment
- when changing the concentricity, ensure all four screws are tight to start, then loosen them very slightly.

b. - the three screws used to level the baseplate may be worn and slipping, or the steel under the screws may be rusted
- turn the MAIN power off

- remove the three large screws and the three small screws in the motor base plate
- remove the motor from the housing, and place it beside the test station.
- clean the small plates that the leveling screws touch using acetone, then polish with some crocus cloth
- reinstall the motor
- replace the three small screws, if they are worn or dirty
- recheck the alignment.

c. - if the four top screws (for concentricity) and the three bottom screws (for flatness) are not tightened properly, the alignment may shift
- each screw should be tightened slowly, while monitoring the alignment on the dial guage.

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TABLE I

SETTINGS FOR CALIBRATION OF TORQUE AND NORMAL OUTPUTS

	Torque	Normal
Motor	ON	ON
Temperature	700°C	25°C (optional)
Environmental Chamber	OFF	ON (optional)
Rate	0.9 rad/sec	0.9 sec^{-1}
% Strain	10%	-
Mode	rate sweep	rate sweep
Geometry	cone and plate or parallel plate	cone and plate or parallel plate
Test type	dynamic	steady (CW or CCW)
Enable Plot	OFF	OFF
Enable Print	OFF	OFF
Sweep Parameters	Last rate 100 points per decade 5	Last rate 100 points per decade 5 readings per rate 1
Initiate	START, HOLD	START, HOLD
Output readings	on TORQ	on NORM

TABLE II

CALCULATED MINIMUM VOLUMES REQUIRED FOR VARIOUS CONE/PLATE FIXTURES (R = 2.5cm)

Cone Angle (radians)	Cone Angle (degrees)	h[a] (cm)	Volume of Cone (cm^3)[b]	Volume of Cylinder (cm^3)[c]	Minimum Fluid Volume (mL)[d]
0.01	0.573	0.025	0.164	0.491	0.327
0.02	1.146	0.050	0.327	0.982	0.655
0.04	2.292	0.100	0.655	1.963	1.308

a h = R x tan (cone angle)
b Cone Volume = 1/3 Π R^2 h
c Cylinder Volume = Π R^2 h
d Fluid Volume = Cylinder Volume - Cone Volume

TABLE III

INTERNAL CALIBRATION OF THE TORQUE AND NORMAL FORCES

CALIBRATION	LOCATION	TRANSDUCER SELECT POSITION	CALIBRATION POT
Torque	Signal Conditioner drawer, 3rd board from the front	1	R13
		10	R10
		100	R16
Normal Force	Signal Conditioner drawer, 4th board from the front	1	R13
		10	R10
		100	R16

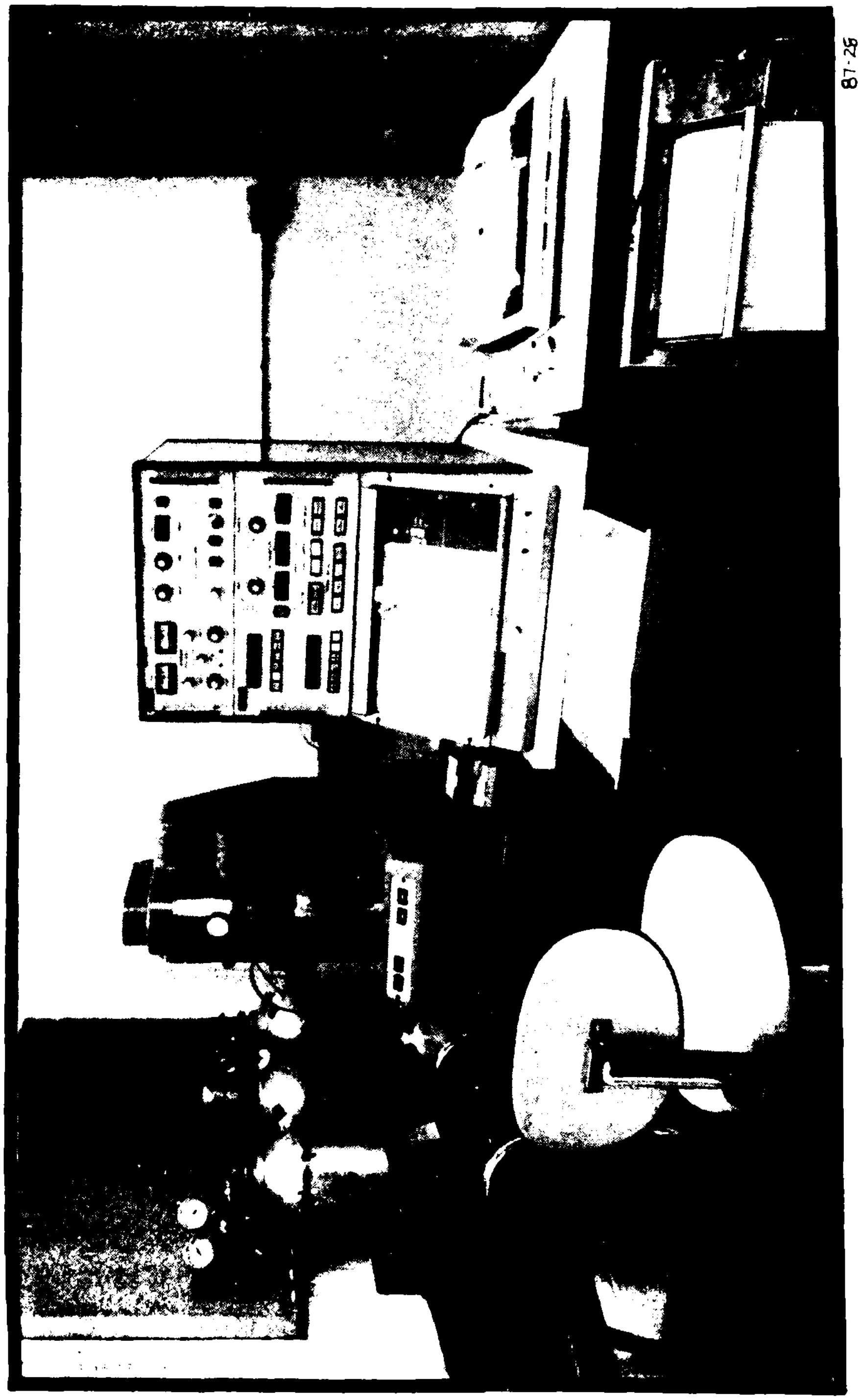

Figure 1
RFR SETUP AT DRES

LEGEND FOR FIGURE 2

Schematic of RFR Test Section

Part Number	Description
2 – 1	Gap indicator gauge
2 – 2	Course gap set indicator
2 – 3	Course spindle position control
2 – 4	Fine spindle position control
2 – 5	Thermocouple plug-in
2 – 6	Fine gap set indicator
2 – 7	Environmental chamber
2 – 8	Sample cup
2 – 9	Connections for circulator bath
2 – 10	Pump reset switch
2 – 11	Environmental chamber switch
2 – 12	Heater indicator light
2 – 13	Motor switch
2 – 14	Main power switch
2 – 15	Motor unit
2 – 16	Transducer assembly
2 – 17	Transducer spindle lock
2 – 18	Gap position stop
2 – 19	Attachment for air line
2 – 20	Transducer shaft

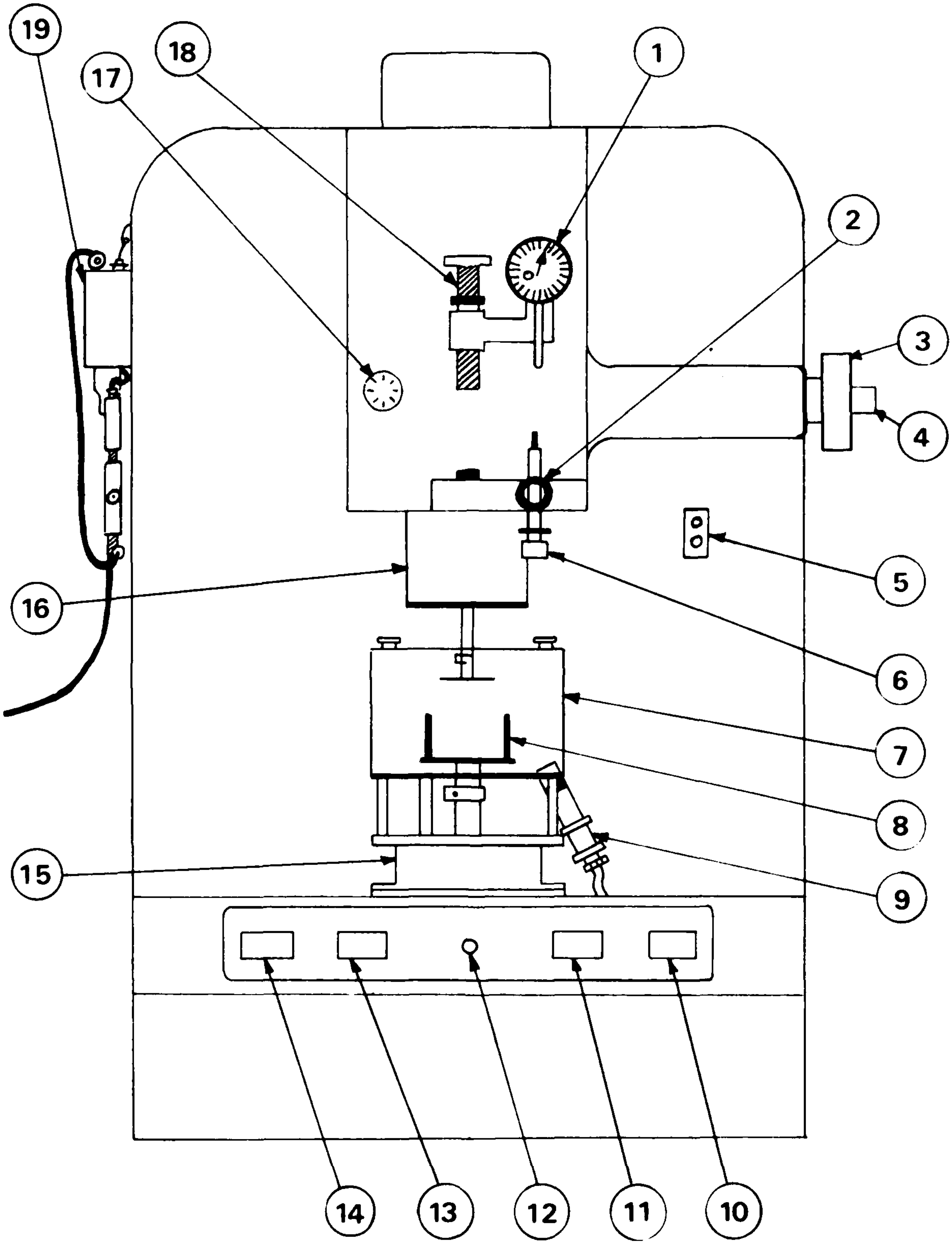

Figure 2

SCHEMATIC OF RFR TEST STATION

LEGEND FOR FIGURE 3

Schematic of RFR Signal Conditioner

Part Number	Description
3 – 1	Course offset adjusts for toruqe and normal force meters
3 – 2	Fine offset adjusts for torque and normal force meters
3 – 3	Torque and normal force calibration screws
3 – 4	Transducer switch
3 5	Torque and normal force meters
3 – 6	Auxillary outputs Y output selector switch
3 – 7	Auxillary outputs Y gain selector switch
3 8	Auxillary outputs X sweep selector switch
3 – 9	Auxillary outputs start/reset switch
3 – 10	Strain servo controller on/off switch
3 – 11	Strain servo controller offset switch
3 – 12	Strain servo controller direction control switch
3 – 13	Strain servo controller input switch
3 – 14	Strain servo controller dynamic/steady selector switch

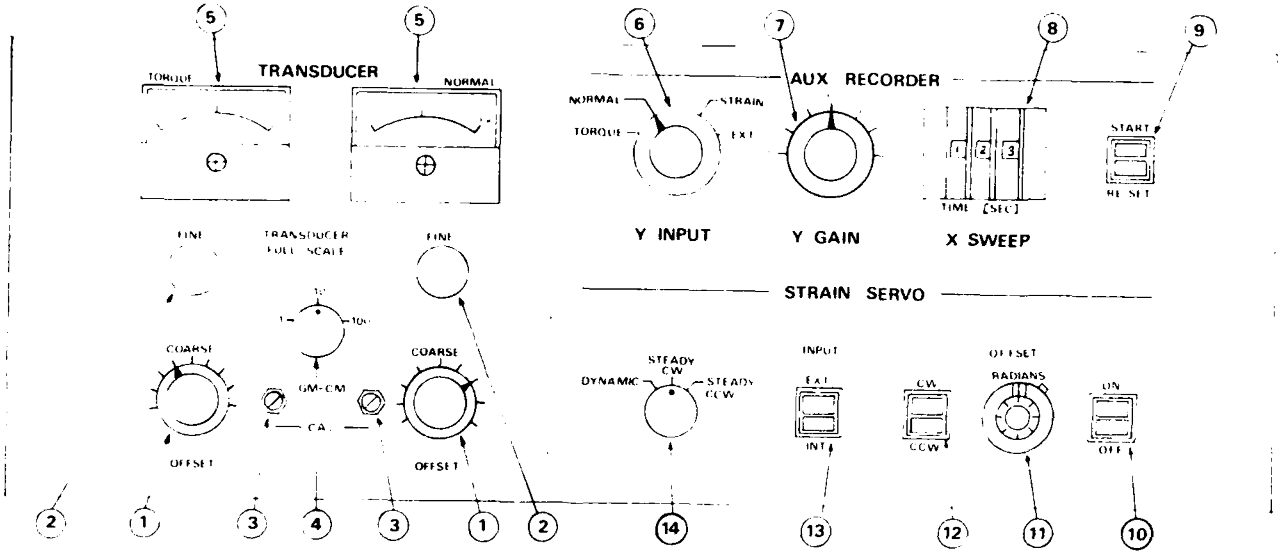

Figure 3

SCHEMATIC OF RFR SIGNAL CONDITIONER

LEGEND FOR FIGURE 4

Schematic of RFR Computer Controls

Part Number	Description
4 – 1	Data select switches
4 – 2	Digital displays
4 – 3	Bath indicator light
4 – 4	Mode select switch
4 – 5	Test select switch
4 – 6	Temperature set switch
4 – 7	Rate set switch
4 – 8	Stain set switch
4 – 9	Output select switch
4 – 10	Sweep parameter selector switch
4 – 11	Test geometry select switch
4 – 12	Output control switches
4 – 13	System control switch
4 – 14	System control switches
4 – 15	Status indicator displays

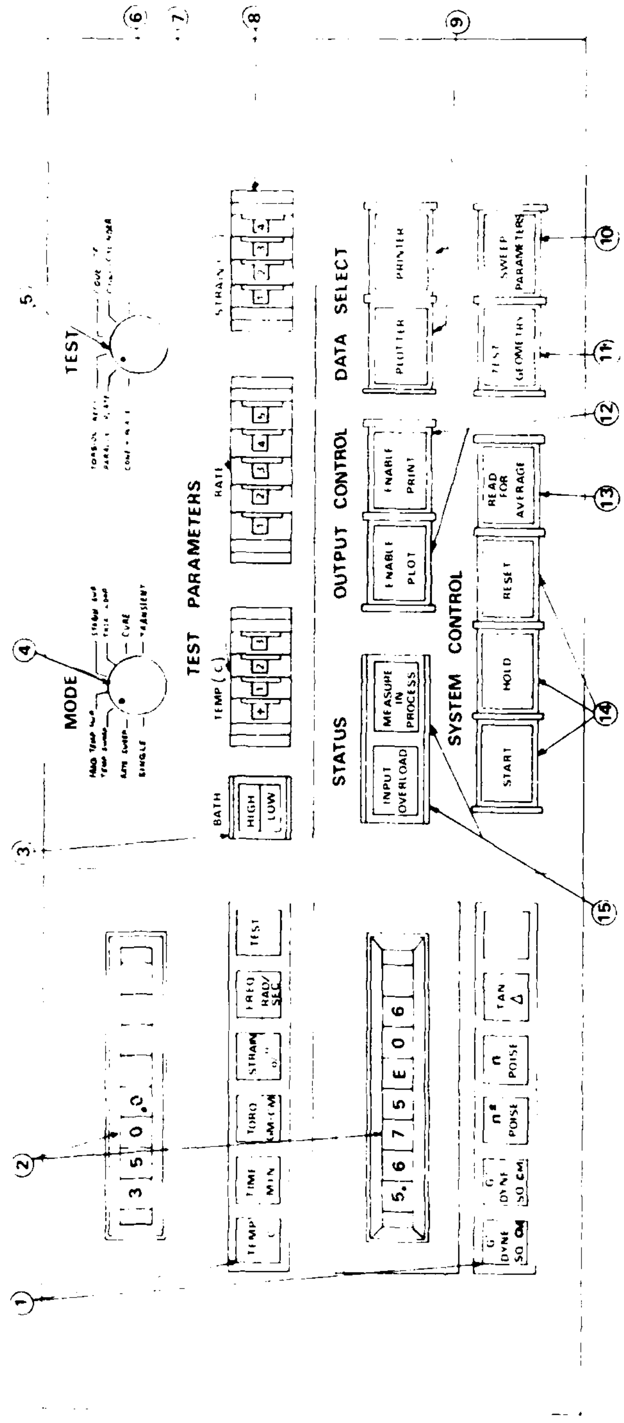

Figure 4

SCHEMATIC OF RFR COMPUTER CONTROLS

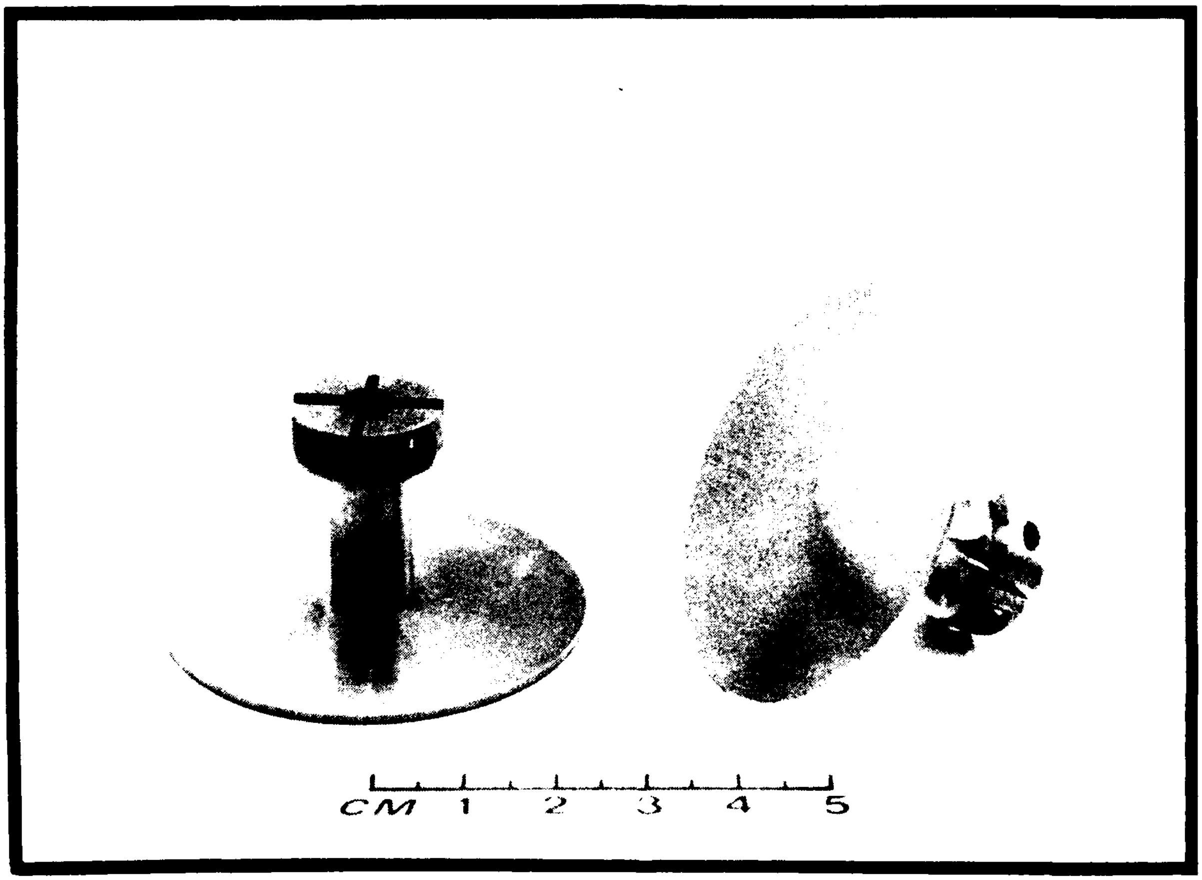

Figure 5

TITANIUM CONES

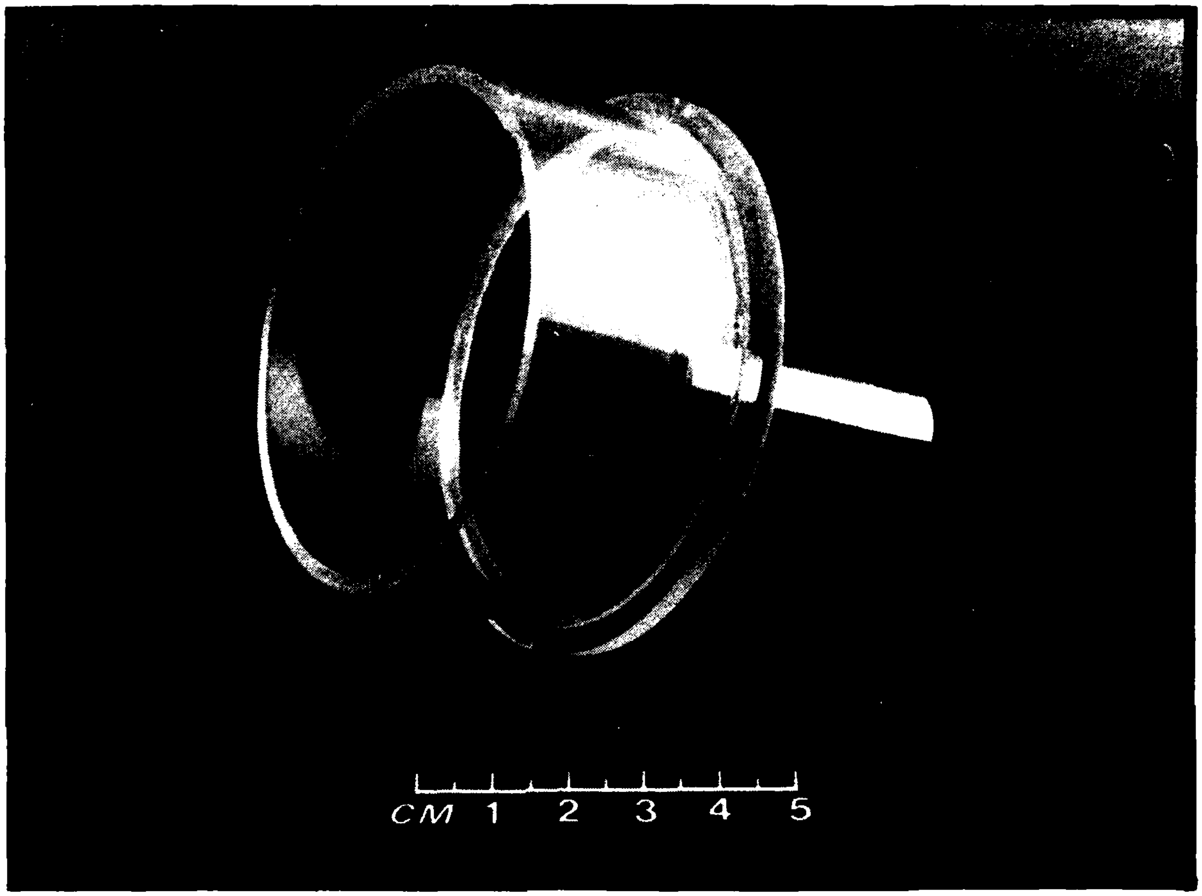

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Figure 6

GLASS SAMPLE CUP

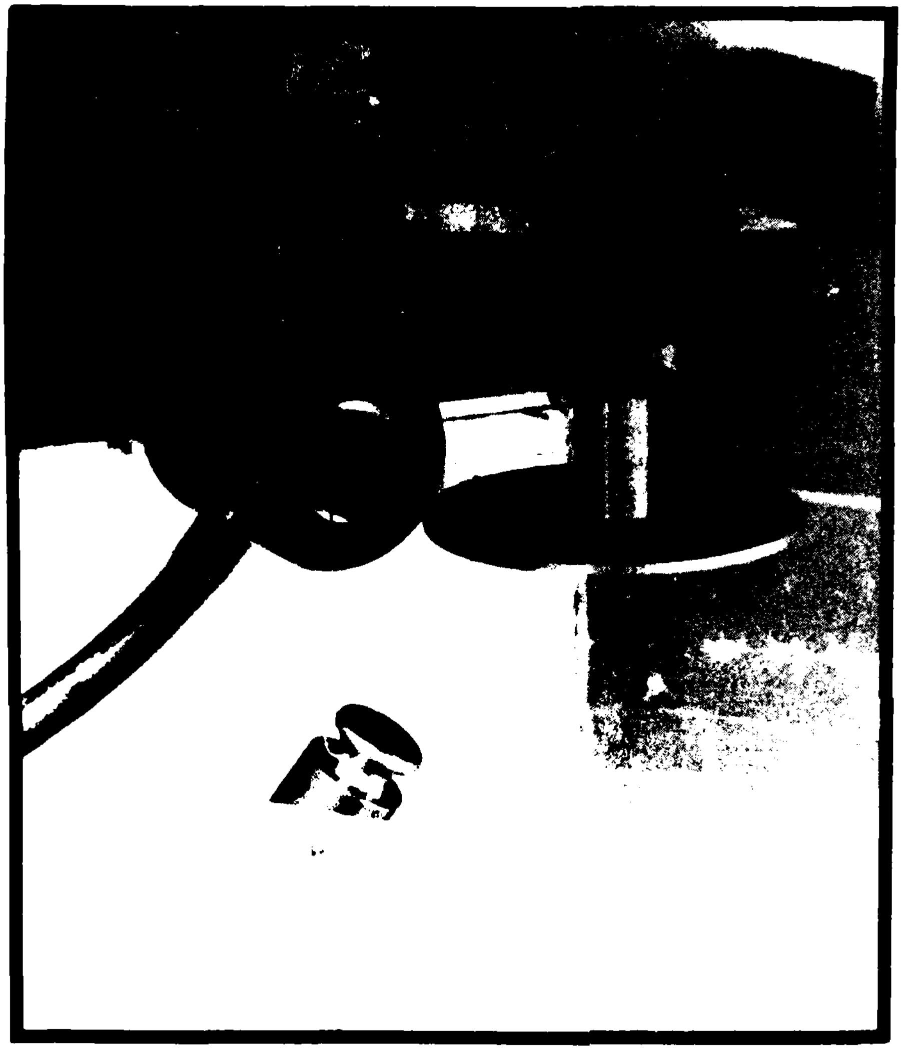
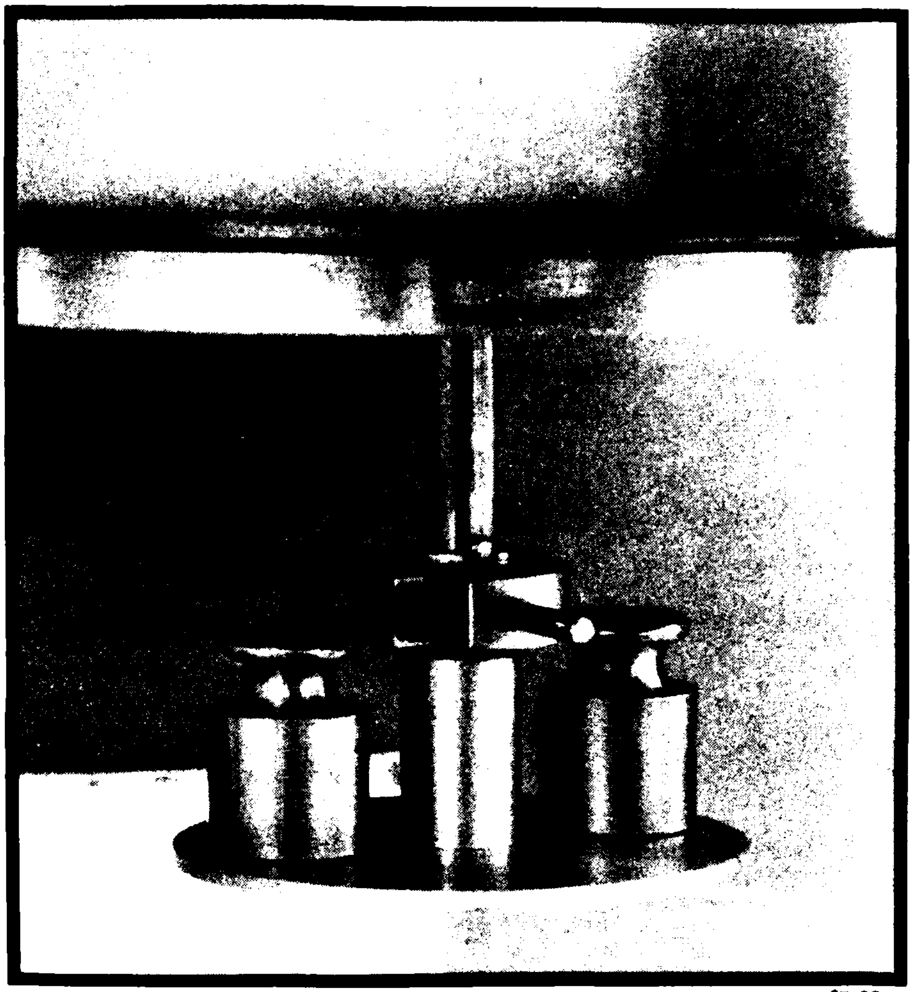


Figure 7

TORQUE CALIBRATION FIXTURE AND AIR PULLEY



87-28

Figure 8

NORMAL FORCE CALIBRATION

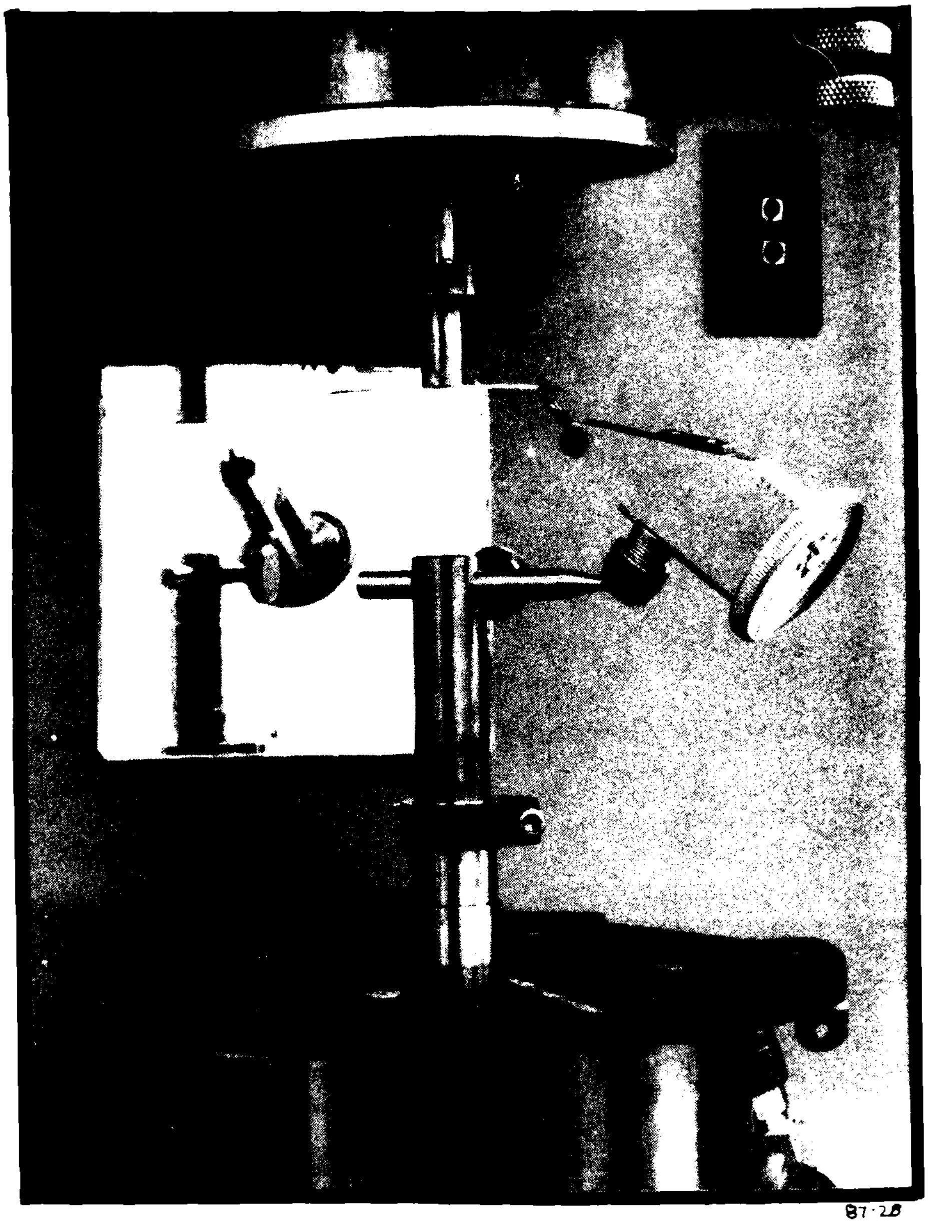


Figure 9

SET UP FOR CONCENTRICITY AND FLATNESS CHECK

APPENDIX I

Specification Sheets for Rheometrics

Fluids Rheometer Fixtures

RHEOMETRICS, INC.

CERTIFICATION

SYSTEM: [illegible]

DATE: [illegible]

MATERIAL: [illegible]

INSP: [illegible]

S/N: [illegible]

CUST: [illegible]

NOMINAL CONE ANGLE [illegible] (RAD)

NOMINAL GAP: [illegible] (mm)

MEASURED CONE ANGLE (ACTUAL) [illegible] (RAD)

ACTUAL GAP: [illegible] (mm)

Q.C./AUTH. [illegible]

RHEOMETRICS, INC.

CERTIFICATION

SYSTEM: RF 2

DATE: 0/26/82

MATERIAL: [illegible] Cone ([illegible]) (Short)

INSP: [illegible]

S/N: 133

CUST: [illegible] (CANADA)

NOMINAL CONE ANGLE .040 (RAD)

NOMINAL GAP: .050 (mm)

MEASURED CONE ANGLE (ACTUAL) .0396 (RAD)

ACTUAL GAP: .0503 (mm)

Q.C./AUTH. [illegible]

RHEOMETRICS, INC.

<u>CERTIFICATION</u>

SYSTEM: RFR

DATE: 10/29/85

MATERIAL: 50mm Cone (short shaft)

INSP: Q.C. 5

S/N: 117B

CUST: NRC

NOMINAL CONE ANGLE 0.02 (RAD)

NOMINAL GAP: 0.050 (mm)

MEASURED CONE ANGLE 0.0201 (RAD)
(ACTUAL)

ACTUAL GAP: 0.050 (mm)

Q.C./AUTH. INSPECTED
U.G. BY 10/29/85 DATE

ONE POSSUMTOWN ROAD, PISCATAWAY, N.J. 08854—TELEPHONE 201/560-8550 TELEX: 219107 RHEOUR—FACSIMILE: (201/560-7451)

RHEOMETRICS, INC.

CERTIFICATION

SYSTEM: RFR

DATE: 10/24/85

MATERIAL: 50mm cone ([illegible])

INSP: Q.C. 5

S/N: 117B

CUST: NRC

NOMINAL CONE ANGLE 0.02 (RAD) NOMINAL GAP: 0.050 (mm)

MEASURED CONE ANGLE 0.0201 (RAD) ACTUAL GAP: 0.050 (mm)
(ACTUAL)

Q.C./AUTH. INSPECTED [illegible] BY 10/24/85 DATE

RHEOMETRICS, INC.

CERTIFICATION

SYSTEM: R.F.R. DATE: 7/14/82

MATERIAL: 50 mm CONE (T.) INSP: [illegible]

S/N: 117 CUST: N.R.C.

NOMINAL CONE ANGLE .020 (RAD) NOMINAL GAP: .050 (mm)

MEASURED CONE ANGLE (ACTUAL) .018 (RAD) ACTUAL GAP: .046 (mm)

Q.C./AUTH. [illegible signature]

60 Fadem Road, Springfield, N.J. 07081 • Telephone 201/467-4700

APPENDIX II

RHEOMETRICS FLUIDS RHEOMETER
REPORT SHEET

DATE:

OPERATOR:

WEIGHT (g.cm)	TORQUE (g.cm)	VOLTAGE (v)	WEIGHT (g)	NORMAL FORCE (g)	VOLTAGE (v)

REMARKS:

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This Sheet Security Classification

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Rheometrics Fluid Rheometer

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Suffield Special Publication No. 105

5. AUTHOR(S) (Last name, first name, middle initial)

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13 ABSTRACT

The Rheometrics Fluids Rheometer (RFR) is a sophisticated, automated device for measuring the rheological properties of low to medium viscosity fluids. This publication is a detailed users manual for the RFR. It contains information on the instrumentation, daily operations and special procedures for the RFR. It should be used in conjunction with the Operations Manual for the RFR supplied by the manufacturer - Rheometrics, Inc.

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KEY WORDS

rheometer
manual
maintenance

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